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TEMPERATURE VARIATIONS OF SPECIMENS IN CRYOGENIC IMPACT TESTING

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This paper reports the temperature variations of specimens in different sizes (10 x 10 x 55 mm, 7.5 x 10 x 55 mm and 5.0 x 10 x 55 mm), which were measured 3 and 5 seconds following their removal separately from a bath at three cooling temperatures (213°, 173° and 77°K) to 293°K atmosphere (placed on an anvil of testing machine). The specimens were 1Cr18Ni9Ti austenite stainless steel, #45 steel, high-quality carbon steel, and Al. The temperature variations of 15Mn26Al4 steel were measured at 3- and 5-second intervals after the specimen was moved from temperatures 20° to 293°K.

A simple temperature-maintaining method was proposed (from 20° to 293°K) for specimens undergoing impact tests at any temperature.

Derived were temperature variation curves for Al specimens during impact tearing tests at 213° and 173°K.

I. Foreword

The application of cryogenic technique has been more and more widespread following the development of science and technology. One of the important indexes of toughness and brittleness of materials during cryogenic equipment design is the

impact energy (kg-m). At present, this index is extensively referred to both at home and abroad. However, there are still few devices used exclusively to measure materials' cryogenic impact energy A_k. Especially when the testing temperature is below 77°K, more difficulties are confronted by the testing researchers. Cryogenic impact testing machines [1, 2] were developed and manufactured; however, these machines did not meet consumer demand because of high price, narrow temperature range (during testing), or low accuracy in temperature control.

At present, standard methods of cryogenic impact tests are confined to temperatures equal to or higher than 77°K. As specified in Metal Material Tests—Impact Toughness Tests (DIN50115-75), for example, the allowable deviation is ± 2°C between the testing temperature and the temperature reached by the temperature-adjusting device while tests are conducted between +55°C and -196°C. If the testing temperature does not fall within this range, or the specimen dimensions are different from those specified, a blank test should be conducted first in order to obtain the proper testing conditions (temperature and the required time interval after the testing specimen is removed from the temperature-adjusting device to the time of testing). The required temperature of the specimen is maintained during tests. In the ASTM standard method, the allowable deviation from the environmental temperature is specified while conducting the test (77°K) on a specimen. As specified in the YB19-64 method, the impact testing specimens should have 3° to 4° supercooling for tests between the room temperature and 213°K. For impact testing conducted at a temperature lower than 213°K, the supercooling should be between 4° and 6°. For specimens undergoing tests (2 to 5 seconds) after being cooled in boiled liquid nitrogen, this is equivalent to a testing temperature of around 93°K, which is also the temperature of the material characteristics. The above-mentioned standards do not mention how to conduct tests at temperature lower than 77°K. The problem of whether or not the above-mentioned specifications are appropriate can be discussed further; the problem is the concern of all researchers.

We all know that the above-mentioned standard methods of cryogenic impact testing should include the three following steps: first, the specimen is placed in a cooling agent long enough (10 to 15 minutes) to cool the specimen thoroughly. Then, a clamp (cooled thoroughly at the same temperature) is used to pick up the specimen from the cooling agent and to place the specimen on an anvil of the

testing machine. In the final step, the specimen is struck by a swinging hammer. From this procedure, we know that at the instant the hammer touches the specimen, it is not at the pre-selected temperature. In order to determine the characteristics at the required temperature, the exact temperature at the instant the specimen is struck by the swinging hammer should be measured. This requirement is difficult to achieve.

Some researchers [3] measured the temperature change of small aluminum specimens at intervals of 1.5 to 10 seconds as the specimens are moved from 4.2°K, 195°K, and 77°K to room temperature. Some other researchers [4] measured the temperature variations of specimens after they are cooled in liquid hydrogen and then brought to room-temperature atmosphere. They reported that during the first second, the specimen temperature rises at least from 20°K to 30°K. While a specimen is picked out of a liquid-helium bath and placed in room-temperature atmosphere, the specimen temperature during the first second at least rises from 4.2°K to 40°K; during the second second, the specimen temperature rises to nearly 77°K. This shows that the present testing methods cannot be used in impact testing of lower than 77°K. For testing at temperatures higher than 77°K, it is also necessary to study further the specifications as prescribed in the standard methods.

Up to now, the authors have not seen data of (specimen) temperature variations at cryogenic impact testing of ferrous metal and its alloys with thermal conductivity lower, and thermal capacity considerably higher, than that of aluminum. At present, researchers can still assume the temperature of the cooling agent as the required temperature of impact testing. Or, a supercooling value is added to the specimen, thus assuming that the specimen temperature during impact is the required testing temperature in a cryogenic impact test. In reality, because of differences in specimen characteristics, dimensions, testing temperatures, environmental temperatures, and operators' degrees of skillfulness, there should be different values of specimen supercooling. In particular, the specimens thoroughly cooled in boiled liquid nitrogen should not be considered as having characteristics of 93°K after testing. Therefore, it is very necessary to determine values of specimen supercooling according to testing conditions.

II. Materials and Testing Procedures

Testing materials: aluminum, 1Cr18Ni9Ti austenite stainless steel, and #45 high-quality carbon steel; 15Mn26Al4 austenite low-temperature steel was used in 20°K tests.

Testing equipment: DC-01 cryogenic-impact-test automatic isothermal groove [5], etc. At 20°K testing, an AuFe—NiCr thermocouple was used. At tests above 77°K, a NiCr—Cu (soldering) thermocouple was used to measure temperatures. Deviations (of thermocouple) from standard temperature were smaller than ±0.5°K. The warm and cold terminals of the thermocouple were at the same temperature; after picking the warm terminal (of the thermocouple while it is still connecting to the specimen) out of the cold bath, the temperature difference between warm and cold terminals can be read out and recorded from the instrument.

Specimen dimensions: specimens were cut with V- or U-shaped notches at dimensions (width x height x length) of 10 x 10 x 55 mm, 7.5 x 10 x 55 mm, and 5.0 x 10 x 5.5 mm.

Specimen positions during temperature measurement: 2 mm from the notch, drill holes (5 mm, 3.5 mm, and 2.5 mm deep) of 1-mm diameter for different specimen dimensions. Then thermocouples were inserted into holes while maintaining good thermal contacts with the respective specimens before sealing the holes. This can avoid the accumulation of cooling medium in holes affecting accuracy of temperature measurement of the specimen.

After maintaining constant temperature for 15 minutes by placing specimens, thermocouples, and specimen clamp in an automatic isothermal groove, specimens (with the temperature-measuring thermocouples) are picked up and placed on an anvil of the testing machine. The temperature variations of specimens were recorded by an automatic tracing-recording instrument, thus obtaining the variation values at different time intervals.

III. Testing Restuls

Table 1 and Fig. 1 show the temperature variation results of specimens as measured in ranges of 213-293°K, 173-293°K, 77-293°K, and 20-293°K. Figures 2 and 3 show the temperature variations (temperature rise from point A to point B) at impact tearing tests of aluminum specimens at 213°K and 173°K.

Table 1. Temperature rise* (after cooling) of specimens.

(e) 材质	(d) 样品尺寸 (mm) \ (c) 3Sec 和 5Sec 内的温度回升(K) \ (b) 测温条件 \ (a) 温度范围(K)	213—293				173—293				77—293			
		(i) 不保护		(j) 保护		(i) 不保护		(j) 保护		(i) 不保护		(j) 保护	
		3Sec	5Sec	3Sec	5Sec	3Sec	5Sec	3Sec	5Sec	3Sec	5Sec	3Sec	5Sec
(f) 纯铝	10×10×55	≤3	—	≤1	≤2	≤3	≤7	≤2	≤5	≤2	≤4	≤2	≤2
	7.5×10×55	≤3	≤5	≤1	≤2	≤5	≤11	≤2	≤5	≤3	≤6	≤2	≤2
	5.0×10×55	≤3	≤6	≤1	≤2	≤6	≤14	≤3	≤6	≤4	≤7	≤2	≤3
(g) 不锈钢	10×10×55	≤1	≤2	≤1	≤1	≤1	≤2	≤1	≤1	≤2	≤3	≤2	≤3
	7.5×10×55	≤1	≤2	≤1	≤1	≤1	≤2	≤1	≤1	≤2	—	≤2	≤2
	5.0×10×55	≤1	≤2	≤1	≤1	≤1	≤2	≤1	≤1	—	—	—	≤1
(h) 45号钢	10×10×55	≤1	≤2	≤1	≤1	≤1	≤3	≤1	≤2	≤2	≤3	≤2	≤2
	7.5×10×55	≤1	≤2	≤1	≤1	≤1	≤4	≤1	≤2	≤2	≤3	≤2	≤2
	5.0×10×55	≤1	≤2	≤1	≤1	≤1	≤5	≤1	≤3	≤2	≤4	≤2	≤3

* Protection—with medical degreased paper wrapped around a specimen; Without protection—bare specimens. Data of temperature increase of all specimens are the average values of more than three measurements.
Key: (a) Temperature range (°K); (b) Conditions of temperature measurement; (c) Temperature rise (after cooling) in °K within 3 and 5 seconds; (d) Specimen dimensions (mm); (e) Materials; (f) Pure aluminum; (g) Stainless steel; (h) #45 steel; (i) Without protection; (j) With protection.

IV. Thermal Analysis of Temperature Variations in Specimens

Some researchers [4] analyzed the temperature variation of specimens; they concluded that the main reasons of a specimen's rapid temperature rise (while it is placed on an anvil of the testing machine in room-temperature atmosphere after the specimen was thoroughly cooled in low temperatures) are: (1) the environmental

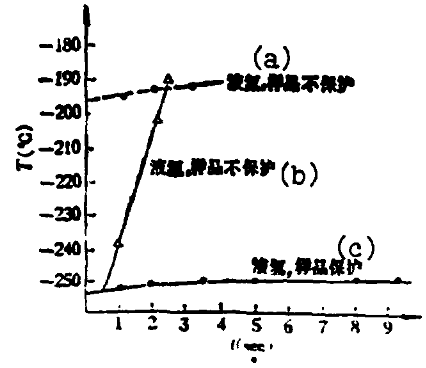

Fig. 1. Temperature variations of 15Mn26Al4 austenite low-temperature-steel (10 x 10 x 55 mm V-shaped notch) specimen with and without protection at 20°K, 77°K and 293°K in stagnant atmosphere.
Key: (a) Specimens (without protection) in liquid nitrogen; (b) Specimens (without protection) in liquid hydrogen; (c) Specimens (with protection) in liquid hydrogen.

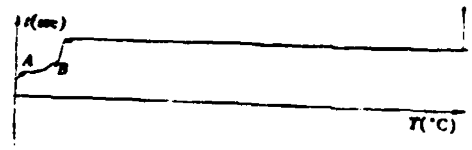

Fig. 2. Temperature variation of 10 x 10 x 55 mm aluminum specimen during impact tearing tests.

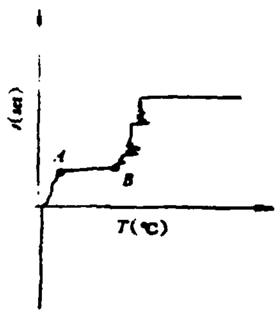

Fig. 3. Temperature variation of 7.5 x 10 x 55 mm aluminum specimen during impact tearing tests.

gases condense at the specimen surface by absorbing heat from the specimen; (2) heat exchange due to collisions between low-boiling-temperature gas molecules and the low-temperature specimen surface as well as the heat radiation on

the specimen by the high-temperature environment; and (3) heat exchange through solid thermal conduction between the low-temperature specimen and the anvil of the testing machine.

While specimens (cooled thoroughly at 77°K, 20°K and 4.2°K) are exposed to room-temperature atmosphere, both water vapor and high-boiling-temperature gases condense on the surface of the cold specimen. Very quickly, many liquid drops of different sizes appear on the specimen surface. After some time, frost forms at the surface of the cold specimen, forming a layer of insulating frost to quickly reduce the rate of temperature rise of the specimen.

Many gases with very low liquefying temperature cannot condense at the surface of cryogenic specimens; however, heat exchange results due to collision between gas molecules and specimen surface. In addition, thermal radiation by the environmental temperature to the specimen cannot be neglected.

The temperature rise of the cryogenic specimen due to solid thermal conduction is determined by the specimen thermal conductivity, anvil temperature (of the testing machine), and time duration of contact.

We all know that the heat capacities of metals and alloys rapidly decrease with temperature reduction; therefore, even a slight thermal disturbance caused by the environment to a cryogenic specimen can result in greater temperature variation.

From the measurement results and thermal analyses mentioned above, the thermal conductivity of aluminum is relatively high and its thermal capacity relatively low; therefore, greater temperature increase results after cooling. In order to stabilize the specimen temperature at 20°K for 3 seconds, 10 cm^3 of liquid hydrogen should be stored around the specimen.

V. Discussions and Conclusions

In order to prevent condensation of high-boiling-temperature gases on the specimen surface, some researchers used the aluminum casing shown in Fig. 4 to enclose the impact testing machine; a gas mixture of hygrogen and helium fills

the casing. Although this method can avoid condensation of high-boiling-temperature gases on the surface of the cryogenic specimen (thus causing a temperature increase of the specimen due to heat exchange of solid thermal conduction, collisions between hydrogen-helium gas molecules and specimen surface, and thermal radiation by the environment to the specimen. Therefore, the temperature of the specimen increases considerably after its cooling.

Some researchers [4] enclosed the specimens in a Meinong [transliteration] paper box as they were thoroughly cooled in liquid hydrogen before testing. The purpose is to preserve a part of the cooling agent in the Meinong paper box in order to insulate the specimens from thermal radiation of the environment and thermal exchange due to collisions between low-boiling-temperature gas molecules and the specimen surface, as well as to prevent condensation of high-boiling-temperature gases on the specimen surface. However, as there is a very limited amount of liquid hydrogen stored in the small box and difficulties arise while centering the specimen on the anvil, very few researchers use this method to maintain specimen temperature.

Fig. 4. Gas-filled casing used for cryogenic impact tests.

In manuscript [6], a testing method of case packaging at 6°K is introduced for recovery of helium. Thus, testing cost is reduced and the problem of temperature increase after specimen cooling is solved. This method has been applied in tests of measuring specimen toughness at low temperatures.

Figure 5 shows the protection of the specimen with medical-absorbent-cotton paper.

Fig. 5. Absorbent cotton for temperature insulation and measurement of specimen temperature in impact tests at 20°K.

At 20°K, the absorbent cotton is very soft so its many pores can store a sufficient amount of liquid hydrogen and its gas at 20°K to keep the specimen from rapidly increasing temperature within a short period of time. Adoption of this method can better deal with three factors of temperature increase (of the specimen) as pointed out in thermal analyses. From Fig. 1, by using the temperature-maintaining method the specimen temperature rises from 20°K to 23°K in 3 seconds; at 9 seconds, the temperature increase does not exceed 5°K. It is both convenient and reliable to maintain (specimen) temperature by using this method. From Table 1, degreased paper is used to protect specimens undergoing cryogenic impact testing at temperatures higher than 77°K. Within 5 seconds, the temperature increase of standard specimens does not exceed 5°K.

Until now, the authors do not know of any paper reporting a standard method of maintaining specimen temperature during cryogenic impact testing.

From thermal analysis, Fig. 1 and Table 1, we can see that the measurement results of specimens are consistent with the above-mentioned discussions whatever the testing temperatures, specimen diemnsions, or materials in cryogenic testing.

From measurement results in Table 1, some standard testing methods specify that the testing results should be further studied while specimens are cooled thoroughly in boiled liquid nitrogen, the whole testing procedures are completed 2 to 5 seconds after specimens are picked out of the cooling agent, and the testing temperatures are about 93°K. In particular, for ferrous metal and its alloys the testing temperature should be 80°K; further studies are required to know whether or not there is consistency between the characteristics of the testing result and the actual characteristics.

From the above discussions, if disregarding differences in specimen materials, dimensions, and testing temperatures, it is irrational to required 4°-6° of supercooling in specimens for tests at temperatures lower than 213°K. We should measure the required (supercooling) degrees according to objective conditions before tests begin.

From Figures 2 and 3, two new surfaces of a specimen result from an impact tearing test; a considerable amount of energy is thus released to rapidly increase the specimen temperature. At a point 2 mm from the new surface, the specimen temperature rose by 7° to 9°. This shows that the impact energy (in macroscopic statistics) in testing is neither the energy at the temperature while tearing at point A, nor the energy while tearing at point B. The energy is the integration of tearing energy of the specimen at temperatures of point A to B. Hence, it is difficult to derive the impact energy of a specimen at a particular temperature. However, this destruction state is consistent to the actual destruction state of the specimen. In other words, during destruction the spreading of tearing causes a temperature increase in the specimen. Therefore, this result still has certain value.

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